UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
NORTHEASTERN REGION
AGRICULTURAL RESEARCH CENTER
BELTSVILLE, MARYLAND 20705

ORIGINAL (Red)

May 24, 1977

Subject: Report of Analysis of Water Samples

To: A. Montague
Environmental Protection Agency, ORD

Following our telephone conversations on the afternoon of May 12, during which I agreed to analyze some water samples for possible chloroform contamination, I prepared two gas chromatographic columns (Pyrex Glass, 6 ft. by 2 mm internal diameter) one packed with Carbowax 20M, the other with Porapak® Q-S. These were conditioned overnight in the usual way. On Friday morning the Carbowax 20M column was installed in our Hewlett Packard Model 7620A gas chromatograph and tested with a chloroform standard in pentane. The standard was made up to contain 9.7 ng of chloroform per µl of pentane. We quickly found that the 20M column was unsuitable for this type of analysis and installed the Porapak® Q column in its place. At a column temperature of 180°C, the Porapak® Q easily resolved water, pentane, and chloroform from each other, the chloroform coming out last with a retention time of approximately 4.2 minutes.

The eight water samples were delivered at approximately noon on Friday, May 13, each sample consisting of approximately 60 ml of water in a septa-sealed vial. We first conducted a rapid screening for gross contamination by injecting 10 µl of each water sample. This analysis did not reveal any evidence for gross quantities of volatile electron-capturing materials. Since our laboratory does not have a liquid sample concentrator such as the Tekmar LSC-1 we proceeded to carry out the analyses by the more conventional techniques utilized for pesticide residues in water. 30 ml of each water sample was removed by a 50 ml capacity syringe and placed in a 250 ml separatory funnel. 5 ml of pentane was then added to each funnel by pipet and the funnels were gently shaken and swirled, venting off the pentane as needed. The water was then drained off and the remaining pentane, approximately 3 to 4 ml, was placed in a small septa-sealed vial. 30 ml of local distilled water was fortified with chloroform to the 100 parts per billion level and analyzed by the pentane extraction technique to determine the percent recovery. Due to variations in the electron capture response to our standard chloroform, calculated recoveries varied from 30 to 74 percent. Nevertheless, the chloroform was distinctly visible in all cases indicating that the method could have detected any chloroform present in the samples at least down to the 100 parts per billion level. No evidence for chloroform was found in any water sample, as reported to you by telephone on Friday P.M.

ORIGINAL
(Red)

A. Montague

On Monday and Tuesday, May 16 and 17, further verification work was carried out using chloroform standards made up in methanol instead of pentane and a freshly prepared, fortified, distilled water sample. The results were essentially the same, confirming our original report to you that no chloroform was present in the samples at least to the 100 parts per billion level. The pentane extraction method used did not permit us to evaluate the presence of methylene chloride, either qualitatively or quantitatively, since the pentane itself eluted ahead of the chloroform at approximately the same retention time as methylene chloride.

Enclosed are xerox copies of some of our chromatograms showing the chloroform signal from our standard solution, a direct injection of water, a pentane extract of the water, and a pentane extract of fortified distilled water.

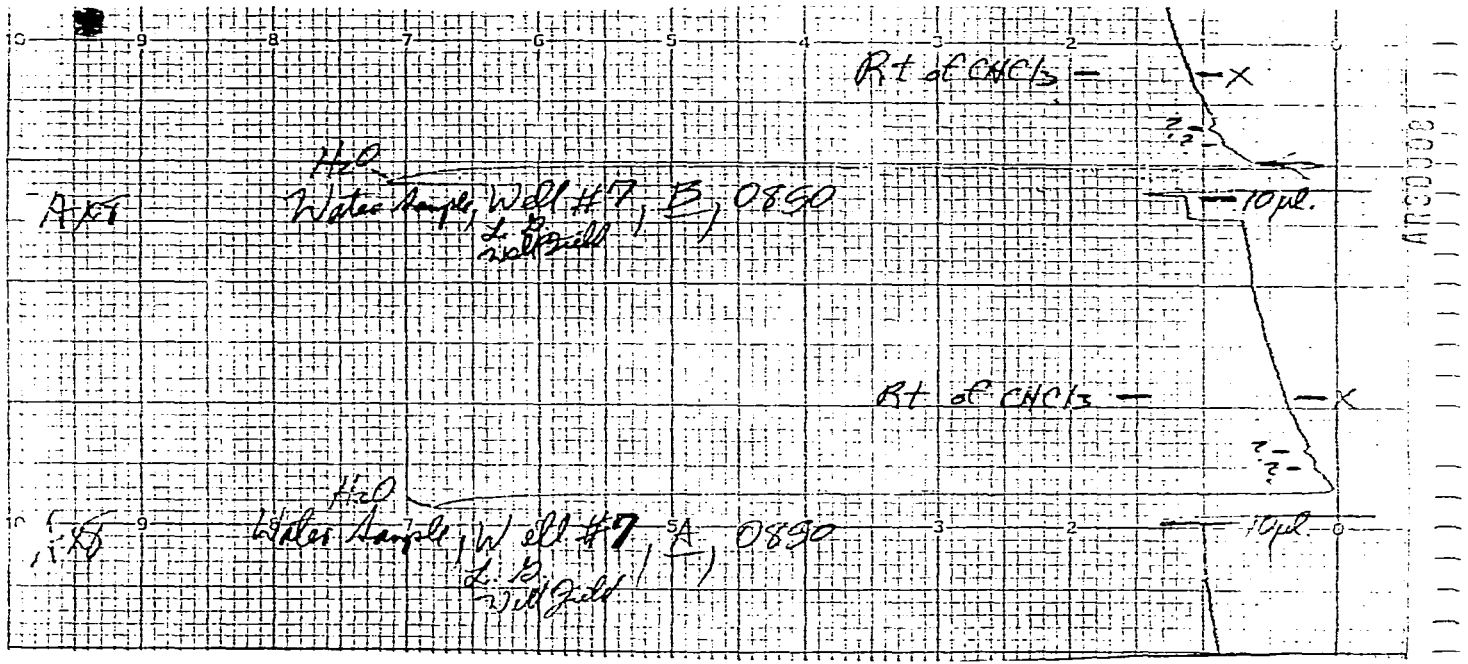
K. R. Hill

K. R. Hill, Chief
Analytical Chemistry Laboratory
Agricultural Environmental Quality Institute

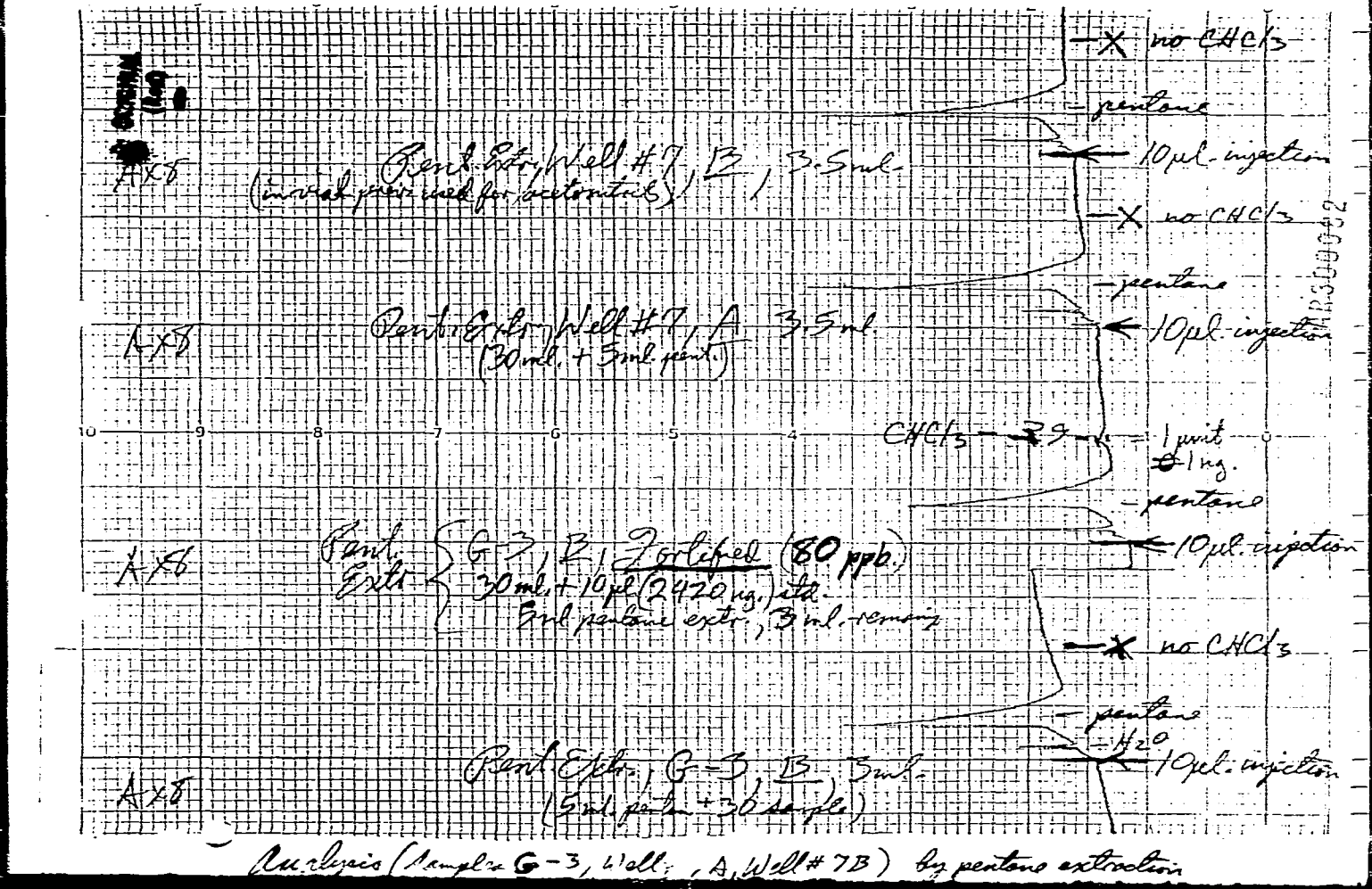
Enclosure

cc:
J. L. Hilton, Chairman, AEQI

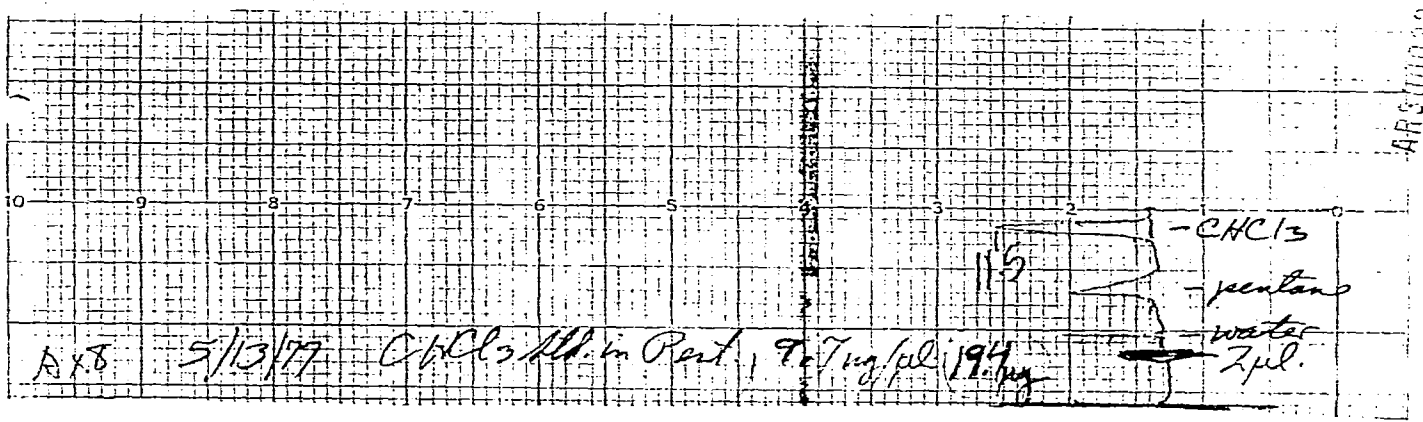
ORIGINAL (Red)



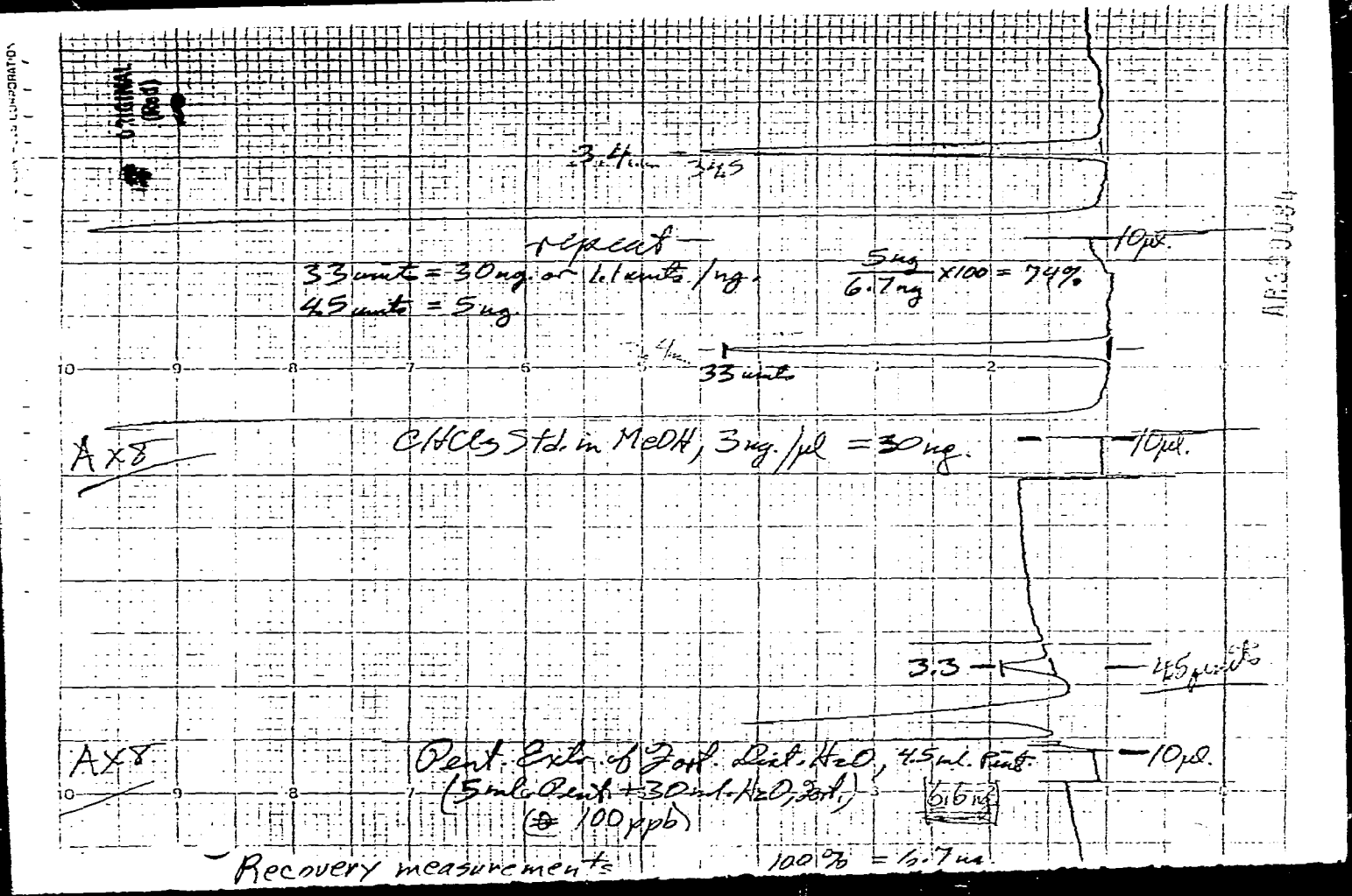
Direct injection of water samples

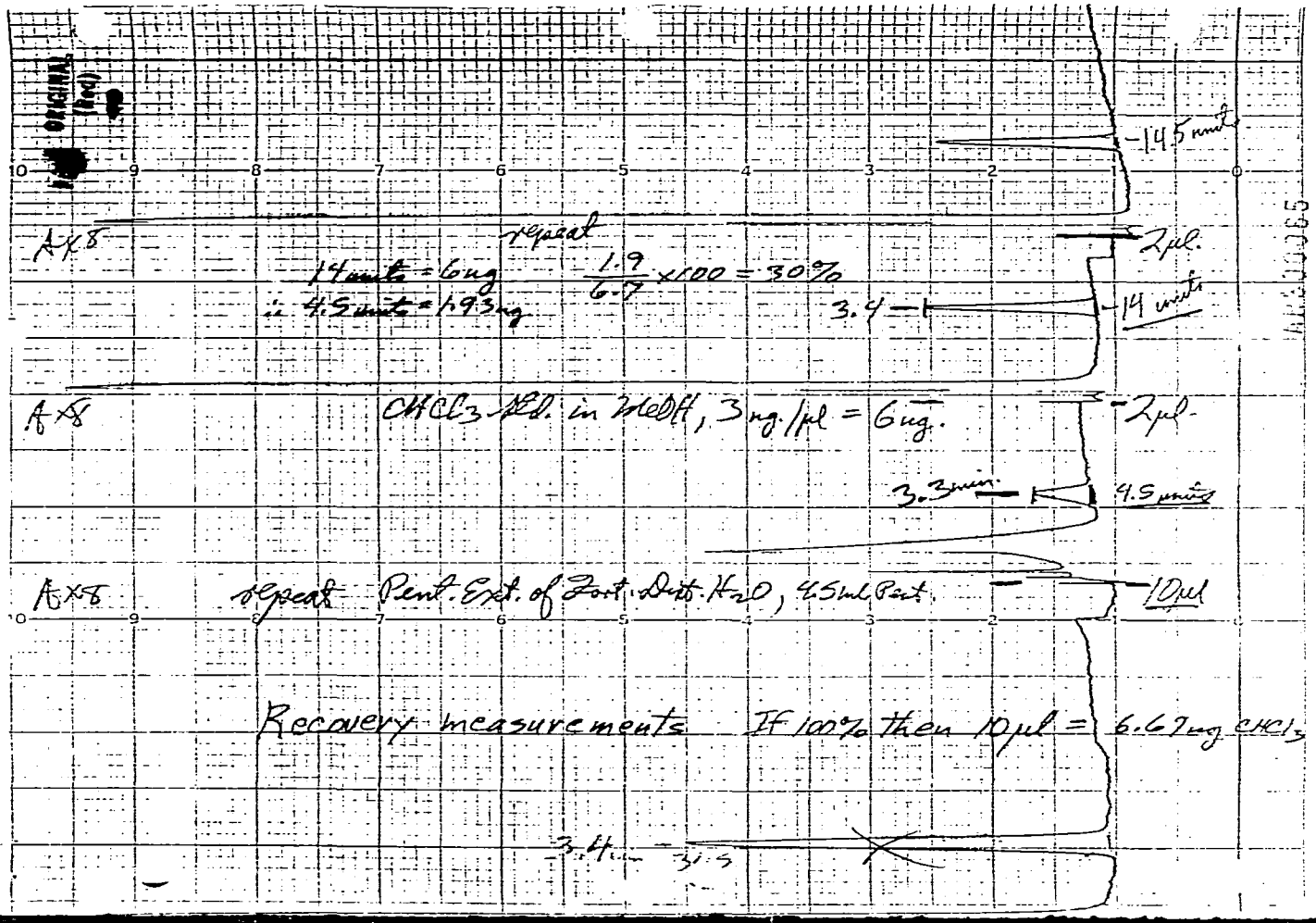


Nucleosis (Sample G-3, Well: A, Well # 7B) by pentane extraction



CHCl3 Standard in pentane


3.4 min
3.45
repeat
10µl.
33 units = 30 ng. or 1.1 units/ng.
45 units = 5 ng.
5ng / 6.7ng x100 = 74%
4 in.
33 units
AX8
C18Cl5 Std. in MeOH, 3 ng./µl = 30 ng.
10µl.
3.3
45 units
AX8
Rent. Extr. of 2 ml. Dist. H2O, 4.5 ml. Rinse
(5 ml. Rent + 30 ml. H2O, 2 ml.)
10µl.
(@ 100 ppb)
6.6 ng
Recovery measurements
100% = 6.7 ng


14.5 units
2μl.
repeat
1.9/6.7 x100 = 30%
14 units = 6ug
∴ 4.5 units = 1.93ug
3.4
14 units
AX8
CHCl3 Std. in MeOH, 3ug/μl = 6ug.
2μl.
3.3min
4.5 units
AX8
repeat Pent. Ext. of Dist. Dist. H2O, 4.5ml Pent.
10μl
Recovery measurements. If 100% then 10μl = 6.67ug CHCl3
3.4
3.5